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THE CRYSTAL AND MOLECULAR STRUCTURE 1-HYDRIDO-1-ISO-PROPYL-

NOV 79 R J RITCHIE , P J HARRIS , H R ALLCOCK N00014-75-C-0685

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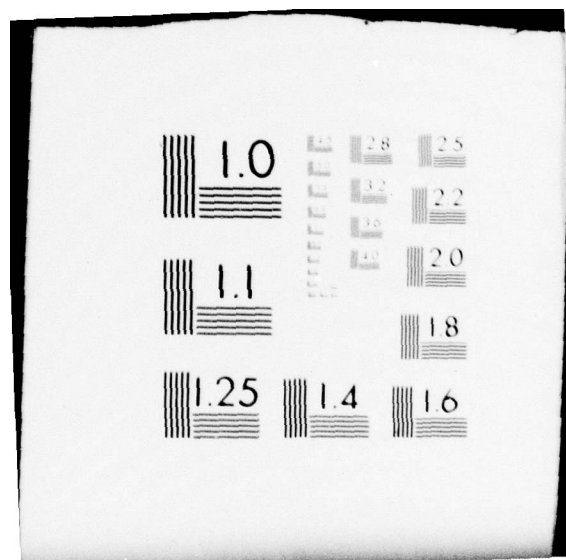
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⁶ THE CRYSTAL AND MOLECULAR STRUCTURE
1-HYDRIDO-1-iso-PROPYL-TETRACHLOROCYCLOTRIPHOSPHAZENE

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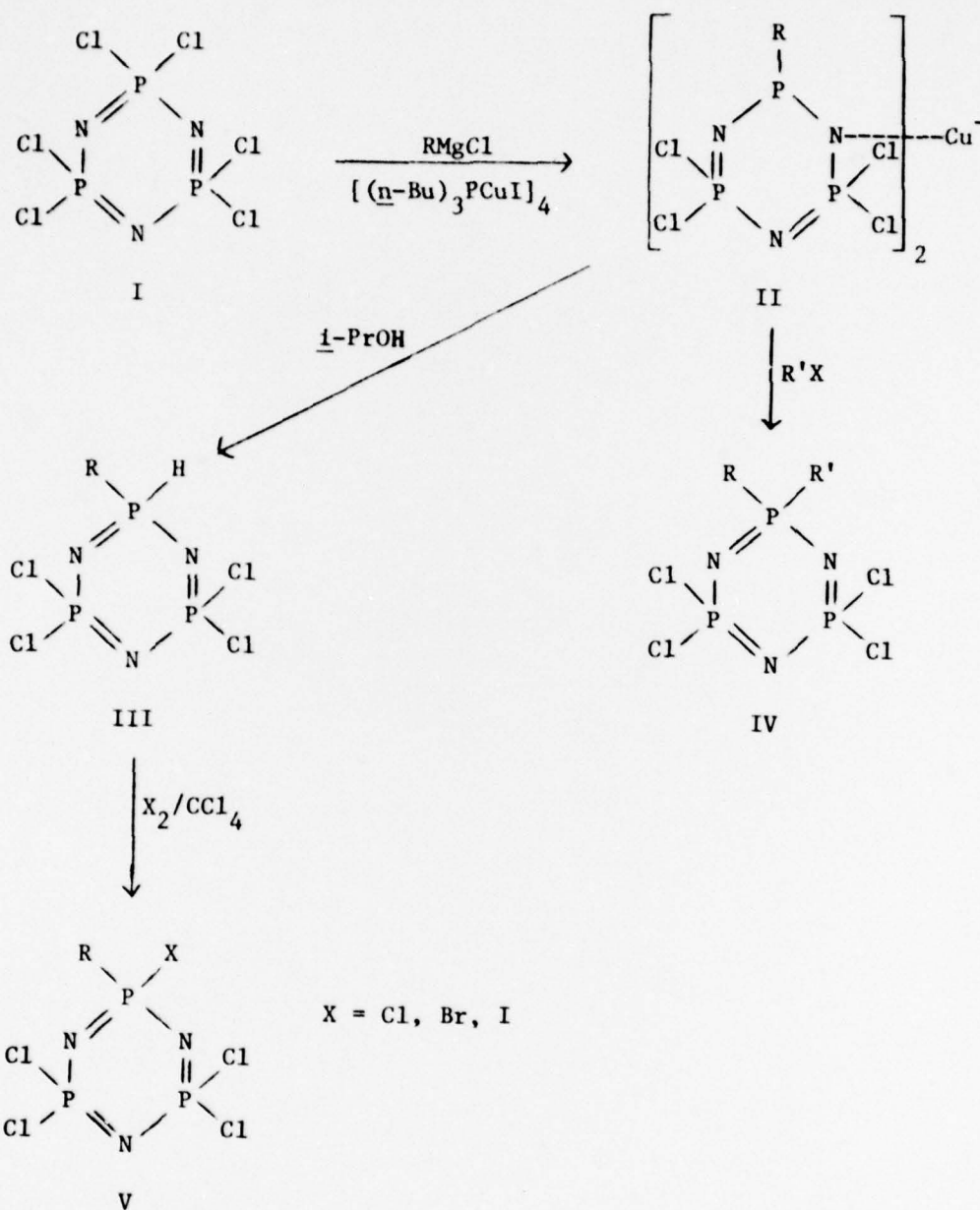
The Crystal and Molecular Structure of
1-Hydrido-1-iso-Propyl-Tetrachlorocyclotriphosphazene.¹

Robert J. Ritchie, Paul J. Harris and Harry R. Allcock*

Contribution from the Department of Chemistry, The Pennsylvania
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ABSTRACT: The first structural determination is described of a cyclic phosphazene, $N_3P_3Cl_4(i\text{-Pr})H$, (VI), which contains both a hydrogen atom and an alkyl group as substituents attached to phosphorus. Compound VI contains a planar phosphazene ring with a curious alternation of P-N bond lengths at progressively greater distances from the P($i\text{-Pr}$)H unit and with corresponding distortions in the bond angles within the ring. The longest P-N bonds are those connected to the P($i\text{-Pr}$)H unit. The phosphorus-hydrogen bond length is $1.26(3) \text{ \AA}$, the phosphorus-carbon bond distance is $1.76(5) \text{ \AA}$, and the H-P-C bond angle is $108.8(7)^\circ$. The structural features of the molecule can be understood in terms of crystal packing forces and π bonding hypotheses. Crystals of VI were orthorhombic with the space group P_{212121} and with $a = 6.128(4) \text{ \AA}$, $b = 11.485(2) \text{ \AA}$, and $c = 17.467(3) \text{ \AA}$, and $Z = 4$. The final R_1 and R_2 factors were 0.043 and 0.045 respectively.



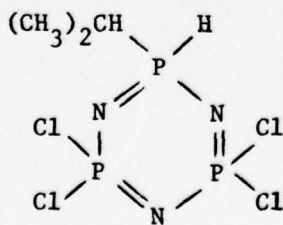
Scheme I

INTRODUCTION

In earlier papers we described the synthesis of a new class of phosphazene compounds that contain a hydrogen atom as a side group bonded directly to phosphorus. These hydrido-alkyl-phosphazenes (III) are synthesized readily via the reaction of hexachlorocyclotriphosphazene (I) with alkyl Grignard reagents in the presence of $[(n\text{-Bu})_3\text{PCuI}]_4$, followed by treatment of the resultant metallo-phosphazene intermediate (II) with *iso*-propanol^{2,3} (Scheme I). Such hydrido-phosphazene compounds (III) have since proved to be important intermediates for the synthesis of a variety of 1-halo-1-alkyl-tetrachlorocyclotriphosphazenes (V),⁴⁻⁶ including those which contain phosphorus-iodine bonds. This approach to phosphazene synthesis via II has also yielded numerous 1,1-dialkylcyclotriphosphazenes (IV) including a number with unsaturated alkyl units.^{7,8}

Scheme I

We describe here the first X-ray structural determination of a hydrido-cyclophosphazene (VI). This particular compound was chosen as a prototype for



VI

structural studies because it is less sensitive to air and moisture than other hydridophosphazenes of this type.^{2,3} The structural determination was carried

out in an attempt to answer the following questions. (1) Is the hydrogen atom linked directly to a phosphorus atom? (Earlier infrared and NMR evidence had provided a strong indication of the presence of a direct P-H bond,^{2,3} but confirmatory evidence was needed). (2) Does the small steric size of a hydrogen atom compared to, say, chlorine or alkyl induce unusual changes in the geometry of the groups attached to phosphorus? (3) What effect, if any, does the hydrogen atom exert on the lengths of the phosphorus-nitrogen bonds, or on the overall geometry of the molecule? (4) Is the hydrogen atom in VI especially sterically shielded in a way that would explain its higher than expected stability to air and moisture?

EXPERIMENTAL SECTION

Synthesis of 1-Hydrido-1-iso-propyl-tetrachlorocyclotriphosphazene (VI).

This compound was prepared by the reaction of hexachlorocyclotriphosphazene (I) with *i*-PrMgCl, in tetrahydrofuran, in the presence of $[(n\text{-Bu})_3\text{PCuI}]_4$, followed by treatment of the intermediate II ($R = i\text{-Pr}$) with *i*-PrOH, as reported earlier.^{2,3} Crystals of VI suitable for single crystal X-ray diffraction were grown slowly from a saturated solution in hexane at -10°C .

Crystallographic Data Collection. A clear fragment of VI, with dimensions ca. 0.4 x 0.4 x 0.5 mm, was mounted on a glass fiber and was placed in a glass capillary tube under an atmosphere of dry nitrogen. Precession photographs were obtained with this crystal with the use of zirconium-filtered molybdenum $K\alpha$ radiation (λ 0.71069 Å). These photographs indicated that the crystal was orthorhombic (Laue symmetry mmm) with the spindle axis parallel to the crystallographic c axis. The observed systematic presences: $h00$, $h = 2n$; $0k0$, $k = 2n$; $00l$, $l = 2n$, gave $P_{2_1^2_1^2_1}$ as the space group. A second crystal, with the dimensions ca. 0.2 x 0.2 x 0.25 mm, was mounted as before and was placed on an Enraf-Nonius CAD-4 diffractometer, controlled by PDP8/a computer. Molybdenum X-rays and a graphite monochromator were used. Twenty-five reflections were located and centered using the Enraf-Nonius

program SEARCH. A least squares refinement of the 2θ values for these twenty-five reflections gave $a = 6.128(4)$, $b = 11.485(2)$, $c = 17.467(3)$ Å, $V = 129.2$ Å³; $d_m = 1.710$ g cm⁻³ (by flotation in tribromomethane/tetrachloromethane mixtures), $d_c = 1.734$ g cm⁻³, $Z = 4$. The calculated molecular weight was 320.92. (Theory for VI = 320.886).

Intensity data were collected using a θ - 2θ scan mode for all reflections where $0.01^\circ \leq 2\theta \leq 60^\circ$. Scan widths were determined with the use of the formula $(SW) = A + B(\tan \theta)$, where A is estimated from the mosaic character of the crystal and B accounts for the increase in the width of the peak due to the $K\alpha_1$ and $K\alpha_2$ splittings. The values determined for A and B were 0.65 and 0.35 respectively. An increase in the scan angle of 25% on each side of the reflection was employed for measurement of background intensities. The net intensities, I_{\sim} , were calculated with the use of the formula $I_{\sim} = CTS - 2(BGR + BGL)$, where CTS defines the total number of counts and BGR and BGL are the right and left background counts respectively. Reflections for which the ratio $\sigma(I)/I_{\sim} < 2$ were labelled as weak and were automatically rejected by the computer. Three check reflections, measured every 1 h, showed no significant change in intensity throughout the entire data collection. The data were corrected for Lorentz and polarization effects, but no absorption correction was applied. The linear absorption coefficient μ , for Mo $K\alpha$ radiation, was calculated to be 13.04 cm⁻¹. Of the 3089 reflections measured, 784 had an intensity greater than $3\sigma(I)$, 896 $> 2\sigma(I)$ and 1084 $> 1\sigma(I)$. Only those reflections for which $I_{\sim} > 3\sigma(I)$ were used in the refinement.

Solution and Refinement of the Structure. The atomic scattering factors used for all the atoms were those of Cromer and Waber.⁹ The function minimized in the least squares refinement was $\sum \omega (|F_o| - |F_c|)^2$, where $\omega = 1/\sigma(F_o)^2$. The residuals R_1 and R_2 are expressed by the formulae $R_1 = \sum |F_o| - |F_c| / \sum |F_o|$, and $R_2 = \left[\sum \omega (|F_o| - |F_c|)^2 / \sum \omega (F_o)^2 \right]^{1/2}$. A three dimensional Patterson synthesis was

used to locate the position of one phosphorus atom. Three cycles of refinement of the positional and isotropic thermal parameters of this atom gave $R_1 = 0.46$. A difference Fourier allowed the location of the other two phosphorus atoms in the ring. Three cycles of refinement on these three atoms lowered R_1 to 0.44. Another difference Fourier map revealed the positions of two of the chlorine atoms. Refinement through three cycles with these five atoms gave $R_1 = 0.34$. A further difference Fourier allowed the location of the other two chlorine atoms and the three ring nitrogen atoms. Three more cycles of refinement on these ten atoms lowered R_1 to 0.16. Another difference Fourier revealed the positions of the three carbon atoms. Refinement through three more cycles lowered R_1 to 0.10. A final difference Fourier allowed the location of all the hydrogen atoms. The introduction of the hydrogen atoms to the model, combined with a change of the thermal parameters to anisotropic for all non-hydrogen atoms, followed by three final cycles of refinement, gave $R_1 = 0.043$ and $R_2 = 0.045$. The isotropic thermal parameters for all the hydrogen atoms were fixed at 1.0 \AA^2 more than the isotropic thermal parameters of the atom to which they were bonded.

The refined positional and anisotropic thermal parameters for all non-hydrogen atoms are given in Table I. The hydrogen atom positions and their isotropic thermal parameters are shown in Table II. A listing of the observed and calculated structure factor amplitudes for the 784 reflections used in the refinement appear in Table III, which is recorded in the microfilm and microfiche editions.

Computations were carried out with the use of a PDP 11/34 computer. The perspective and stereoscopic drawings were obtained with the use of VERSATEC plotter. The computer programs used were the Enraf-Nonius "Structural Determination Package" and the Enraf-Nonius version of the ORTEP program.

RESULTS AND DISCUSSION

General Structural Features of the Molecule. The X-ray structure determination of VI indicated that the phosphazene ring in this molecule is planar. The hydrogen atom and *iso*-propyl group are bound to one phosphorus atom, P(1), and two chlorine atoms are linked to each of the remaining two phosphorus atoms. The mean P-Cl bond lengths of 1.994 Å and the Cl-P-Cl bond angle of 100.05° are similar to the lengths and angles reported for hexachlorocyclotriphosphazene.¹⁰⁻¹² The general structure of the molecule, together with the numbering system used for all non-hydrogen atoms is shown in Figure 1. A stereoscopic view is shown in Figure 2. The bond distances and angles are listed in Table IV.

Bonding About Phosphorus Atom P(1). The skeletal phosphorus atoms in cyclic phosphazenes usually display a distorted tetrahedral geometry, with a ring angle near 120° and an exocyclic angle near 100°. ¹³ As shown in Figure 1, the bond angles at P(1) in VI are appreciably distorted from this normal arrangement. Specifically, the N-P-N ring angle is narrowed to 115.6° and the exocyclic C-P-H angle is widened to 108.9°. This cannot be a result of a steric interaction between the side groups because of the small size of the hydrogen linked to P(1) and the particular orientation adopted by the *iso*-propyl group. Hence, as will be discussed later, the wide exocyclic C-P-H angle is probably a response (scissoring effect) to the narrow N-P(1)-N ring angle which, in turn, may depend on unusual electronic effects within the phosphazene ring.

The P(1)-H(1) bond length is 1.26(3) Å. This distance is shorter than phosphorus-hydrogen bond lengths reported for phosphines. For example, the P-H bond distance in PH₃ was reported¹⁴ to be 1.44 Å. However, this difference is not considered to be significant because of the lack of precision in the location of hydrogen atoms by the X-ray method. A neutron diffraction study¹⁵ has been used to show that the P-H bond distance in PH₄⁺ is 1.414 Å.

Orientation of the iso-propyl Group. The iso-propyl group bonded to P(1) occupied an unusual orientation. The group was positioned in such a way that carbon atom C(2) was located over the phosphazene ring and C(3) was displaced to the side of an imaginary plane through P(1), C(1), C(2) and N(4). Hydrogen atoms H(21) and H(22) lie adjacent to nitrogen atoms N(6) and N(2) respectively. The distances N(6)···H(21) and N(2)···H(22) are almost exactly the sum of the van der Waals radii for these atoms (2.75 Å). Thus, the orientation of the iso-propyl group could be a response to these van der Waals interactions, or even a consequence of weak hydrogen bonding.

However, it was concluded that crystal packing forces are probably responsible for this conformation. A variable temperature ^{13}C NMR study¹⁶ showed that no particular conformation for the iso-propyl group was preferred in solution, even at temperatures as low as -80°C . The spectrum - a doublet of triplets [P-CH(CH₃)₂] centered at 29.53 ppm, ($J_{\text{PC}} = 90.1 \text{ Hz}$, $J_{\text{PNPC}} = 3.2 \text{ Hz}$) and a singlet [P-CH(CH₃)₂] at 12.93 ppm - did not change over the temperature range of $+40^\circ\text{C}$ to -80°C . Thus the unusual orientation found for the iso-propyl group is a phenomenon of the solid state system and is not an intrinsic characteristic of VI in the solution state. In solution, the iso-propyl group may exert some steric shielding effect on the reactivity of the hydrido function.

The P(1)-C(1) bond length was found to be 1.76(5) Å. This distance is similar to phosphorus-carbon bond lengths that have been determined for other alkyl-halo-cyclophosphazenes (Table V).¹⁷⁻²¹ The C(1)-C(2) and C(1)-C(3) bond lengths, of 1.540 Å and 1.537 Å respectively, are typical for carbon-carbon single bonds.²²

Bonding Within the Phosphazene Ring. Three distinctly different P-N bond lengths were found in the skeletal ring of VI. The two P-N bonds connected to P(1), (the phosphorus atom bearing the hydrogen and iso-propyl group),

were longer (1.61 \AA), than the other P-N bonds. In fact, an **alternation** in longer and shorter bonds existed for the P-N bonds located at increasing distances from P(1). Thus P(1)-N(2) and P(1)-N(6) were 1.61 \AA , N(6)-P(5) and N(2)-P(3) were 1.55 \AA , and P(3)-N(4) and P(5)-N(4) were 1.58 \AA . (This last difference is considered to be significant). The lengths of the P-N bonds furthest from P(1) are very similar to those determined for hexachlorocyclotriphosphazene.¹⁰⁻¹² The variation in P-N bond lengths found in compound VI has been observed for other partially alkylated cyclophosphazenes¹⁷⁻²¹ and it has been ascribed to the influence by the less-electronegative substituents at P(1) on the π -bonding in the ring.^{19-21, 23, 24} We agree with this interpretation.

The unequal bond lengths within the six membered phosphazene ring lead inevitably to distortions in the endocyclic bond angles away from 120° . The decrease in the N(6)-P(1)-N(2) bond angle (to 115.6°) has already been mentioned. However, the N(2)-P(3)-N(4) and N(4)-P(5)-N(6) angles (mean value 120.04°) are close to those found in $(\text{NPCl}_2)_3$ ¹⁰⁻¹² and in $(\text{NPF}_2)_3$.²⁵ The exocyclic Cl-P(3)-Cl and Cl-P(5)-Cl angles (100.05°) are similar to those found in $(\text{NPCl}_2)_3$.¹⁰⁻¹² Comparisons between the structural parameters for VI and for closely related phosphazenes are listed in Table V.

ACKNOWLEDGEMENT. We thank the Office of Naval Research for support of this work through Grant No. N00014-75-C-0685.

SUPPLEMENTARY MATERIAL AVAILABLE. Table III. A complete listing of the observed and calculated structure factor amplitudes (6 pages). Ordering information is given on any current Masthead page.

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Table I

Positional and Thermal Parameters for All Non-hydrogen Atoms

Atom	X	Y	Z	B(1,1)	B(2,2)	B(3,3)	B(1,2)	B(1,3)	B(2,3)
Cl(1)	0.0766(5)	-0.1797(2)	0.1412(2)	0.037(1)	0.0088(2)	0.0045(1)	-0.012(1)	0.0069(7)	-0.0005(3)
Cl(2)	0.5231(5)	-0.2387(2)	0.0732(1)	0.037(1)	0.0069(2)	0.0041(1)	0.004(1)	0.0003(7)	0.0006(3)
Cl(3)	0.8339(6)	0.1180(3)	0.1683(2)	0.037(1)	0.0092(3)	0.0071(1)	-0.007(1)	-0.0078(8)	0.0010(4)
Cl(4)	0.3921(6)	0.2038(3)	0.2258(1)	0.058(2)	0.0104(3)	0.0041(1)	-0.004(1)	0.0044(8)	-0.0055(3)
P(1)	0.3446(5)	0.0888(2)	-0.0014(1)	0.0338(10)	0.0046(2)	0.00295(9)	0.003(1)	-0.0015(7)	0.0007(3)
P(3)	0.3354(5)	-0.1013(2)	0.0958(1)	0.0281(9)	0.0049(2)	0.00241(8)	-0.004(1)	-0.0007(6)	0.0000(3)
P(5)	0.5168(5)	0.1022(2)	0.1444(1)	0.0352(10)	0.0052(2)	0.00252(8)	-0.007(1)	-0.0019(7)	-0.0010(3)
N(2)	0.268(1)	-0.0419(6)	0.0195(4)	0.037(3)	0.0043(6)	0.0025(3)	-0.005(3)	-0.007(2)	0.0006(7)
N(4)	0.445(2)	-0.0275(6)	0.1612(4)	0.050(4)	0.0060(6)	0.0023(3)	-0.016(3)	-0.0001(2)	0.0011(8)
N(6)	0.470(2)	0.1574(6)	0.0653(4)	0.061(4)	0.0038(6)	0.0028(3)	-0.008(3)	-0.005(2)	-0.0005(7)
C(1)	0.504(2)	0.0888(8)	-0.0858(5)	0.049(5)	0.0054(7)	0.0038(4)	-0.013(4)	0.001(3)	0.001(1)
C(2)	0.719(2)	0.0216(11)	-0.0757(7)	0.044(5)	0.0119(12)	0.0059(5)	0.004(5)	0.014(3)	0.001(2)
C(3)	0.368(3)	0.0418(11)	-0.1529(6)	0.071(7)	0.0150(13)	0.0035(4)	-0.001(6)	-0.001(4)	-0.001(1)

Table II

Hydrogen Atom Positions and Isotropic Thermal Parameters

Atom	X	Y	Z	B(1,1)
H(1)	0.16(1)	0.137(7)	-0.015(4)	4.5000(0)
H(11)	0.56(1)	0.159(7)	-0.087(4)	6.0000(0)
H(21)	0.78(1)	0.037(7)	-0.024(4)	6.0000(0)
H(22)	0.65(1)	-0.060(7)	-0.061(4)	6.0000(0)
H(23)	0.83(2)	0.041(7)	-0.125(4)	6.0000(0)
H(31)	0.23(1)	0.075(7)	-0.155(4)	6.0000(0)
H(32)	0.43(2)	0.054(7)	-0.198(4)	6.0000(0)
H(33)	0.35(2)	-0.050(7)	-0.127(4)	6.0000(0)

Table III

VALUES OF 10*FOBS & 10*FCALC FOR N3F3(H)CL4(ISO-C3H7)

H	K	L	FOBS	FCALC	H	K	L	FOBS	FCALC	H	K	L	FOBS	FCALC
0	0	2	485	496	0	3	9	506	504	0	7	3	396	399
0	0	4	1026	1047	0	3	12	439	450	0	7	5	593	590
0	0	6	335	333	0	3	13	465	463	0	7	6	92	84
0	0	8	263	267	0	3	15	501	483	0	7	7	1040	1047
0	0	10	184	190	0	3	27	122	76	0	7	8	242	248
0	0	12	690	683	0	4	0	686	658	0	7	9	104	109
0	0	14	657	659	0	4	1	271	255	0	7	10	149	148
0	0	16	201	186	0	4	3	171	169	0	7	11	202	185
0	0	22	136	109	0	4	4	488	477	0	7	12	122	116
0	1	1	69	74	0	4	5	864	823	0	7	15	187	190
0	1	2	1284	1262	0	4	6	554	553	0	8	0	141	126
0	1	3	214	215	0	4	7	153	146	0	8	1	272	284
0	1	4	547	549	0	4	8	405	403	0	8	2	99	96
0	1	5	1174	1182	0	4	9	305	300	0	8	3	368	373
0	1	6	262	261	0	4	10	116	92	0	8	4	288	287
0	1	7	88	121	0	4	11	166	167	0	8	5	460	456
0	1	8	363	353	0	4	12	199	187	0	8	7	258	281
0	1	9	213	219	0	4	13	297	310	0	8	8	343	346
0	1	11	164	172	0	4	17	130	141	0	8	9	478	490
0	1	12	137	126	0	4	19	123	120	0	8	12	99	110
0	1	13	167	177	0	4	20	214	204	0	8	13	200	200
0	1	14	241	245	0	4	22	119	130	0	9	1	270	272
0	1	15	91	41	0	5	1	396	368	0	9	2	410	413
0	1	17	177	167	0	5	2	286	284	0	9	3	252	243
0	2	1	325	307	0	5	3	156	148	0	9	5	273	287
0	2	2	535	529	0	5	6	448	434	0	9	8	101	121
0	2	3	968	927	0	5	7	365	357	0	9	9	128	136
0	2	4	121	123	0	5	8	157	162	0	9	13	255	260
0	2	5	173	175	0	5	9	347	352	0	9	14	134	130
0	2	6	933	867	0	5	10	239	252	0	10	0	477	472
0	2	7	469	456	0	5	12	226	231	0	10	1	130	131
0	2	8	274	274	0	5	18	194	197	0	10	4	122	116
0	2	9	248	255	0	5	21	127	88	0	10	5	254	260
0	2	10	189	198	0	6	0	700	708	0	10	6	133	126
0	2	11	301	310	0	6	1	394	386	0	10	8	319	317
0	2	12	213	218	0	6	2	550	519	0	10	9	111	109
0	2	13	277	289	0	6	3	459	443	0	10	10	103	99
0	2	14	179	188	0	6	5	624	618	0	10	12	97	103
0	2	20	163	150	0	6	7	537	532	0	10	16	123	115
0	3	1	1222	1189	0	6	9	299	301	0	11	2	154	152
0	3	2	1477	1415	0	6	10	412	409	0	11	3	179	197
0	3	3	216	217	0	6	11	324	318	0	11	6	264	258
0	3	4	192	192	0	6	12	326	329	0	11	11	177	172
0	3	5	96	103	0	6	14	191	181	0	12	0	148	154
0	3	6	155	158	0	6	17	157	160	0	12	2	259	254
0	3	7	656	654	0	7	1	237	232	0	12	5	121	28
0	3	8	101	85	0	7	2	76	74	0	12	11	126	87

Table III (continued)

VALUES OF 10*FOBS & 10*FCALC FOR N3F3(H)CL4(ISO-C3H7)

H	K	L	FOBS	FCALC	H	K	L	FOBS	FCALC	H	K	L	FOBS	FCALC
0	13	2	153	121	1	2	11	157	151	1	5	15	153	176
0	13	3	132	134	1	2	12	241	245	1	5	16	126	141
0	13	4	110	128	1	2	13	148	138	1	5	17	192	182
0	13	5	140	140	1	2	14	449	442	1	5	20	111	119
0	13	7	196	194	1	2	15	225	224	1	5	21	154	168
0	14	0	254	274	1	2	16	89	89	1	6	0	81	79
0	14	4	177	183	1	2	17	207	198	1	6	1	330	330
1	0	1	587	608	1	3	0	238	239	1	6	2	345	353
1	0	2	406	415	1	3	1	793	757	1	6	3	599	605
1	0	3	312	312	1	3	3	572	546	1	6	4	193	194
1	0	4	646	645	1	3	4	435	422	1	6	5	279	272
1	0	5	792	800	1	3	5	94	88	1	6	7	259	258
1	0	6	93	89	1	3	6	485	465	1	6	8	256	256
1	0	7	211	226	1	3	7	677	641	1	6	9	112	85
1	0	8	520	530	1	3	8	241	251	1	6	11	105	96
1	0	9	212	225	1	3	9	241	244	1	6	12	191	199
1	0	10	292	298	1	3	10	141	137	1	6	13	186	167
1	0	11	251	258	1	3	12	276	287	1	6	15	227	221
1	0	14	181	193	1	3	13	232	235	1	6	18	111	104
1	0	15	175	188	1	3	14	216	216	1	6	19	123	113
1	1	0	756	773	1	3	16	199	200	1	7	0	436	445
1	1	1	1050	1035	1	4	0	408	397	1	7	1	369	360
1	1	2	546	538	1	4	1	284	264	1	7	2	334	342
1	1	3	509	508	1	4	2	489	464	1	7	3	123	116
1	1	4	829	837	1	4	3	95	90	1	7	4	408	407
1	1	5	653	656	1	4	4	460	457	1	7	6	174	175
1	1	6	243	245	1	4	5	281	292	1	7	7	178	178
1	1	7	185	186	1	4	6	294	294	1	7	8	334	341
1	1	8	575	583	1	4	7	520	506	1	7	9	272	273
1	1	9	128	134	1	4	8	332	329	1	7	12	99	94
1	1	10	691	711	1	4	9	254	268	1	7	13	226	225
1	1	11	91	104	1	4	10	171	161	1	7	18	107	114
1	1	12	406	410	1	4	11	107	130	1	8	1	220	205
1	1	13	376	379	1	4	12	150	148	1	8	2	188	201
1	1	14	128	115	1	4	15	222	218	1	8	3	511	512
1	1	19	138	123	1	4	19	201	170	1	8	4	241	241
1	1	21	117	98	1	5	1	467	477	1	8	5	410	414
1	2	0	1080	1046	1	5	2	980	959	1	8	6	354	367
1	2	1	469	452	1	5	3	82	71	1	8	7	133	144
1	2	3	840	838	1	5	4	363	360	1	8	8	286	278
1	2	4	607	606	1	5	5	322	321	1	8	11	90	83
1	2	5	1083	1081	1	5	6	260	262	1	8	13	164	155
1	2	6	219	223	1	5	7	605	612	1	8	14	142	138
1	2	7	120	136	1	5	8	139	143	1	8	15	183	170
1	2	8	449	451	1	5	9	179	185	1	9	1	299	301
1	2	9	531	525	1	5	10	509	511	1	9	2	286	281
1	2	10	375	379	1	5	12	304	314	1	9	3	269	282

Table III (continued)

VALUES OF 10*FOBS & 10*FCALC FOR N3F3(H)CL4(ISO-C3H7)

H	K	L	FOBS	FCALC	H	K	L	FOBS	FCALC	H	K	L	FOBS	FCALC
1	9	4	133	153	2	0	6	312	307	2	3	2	278	272
1	9	5	162	156	2	0	8	113	105	2	3	3	260	270
1	9	6	207	202	2	0	9	402	409	2	3	4	358	361
1	9	8	126	131	2	0	10	532	571	2	3	5	690	671
1	9	9	122	127	2	0	11	388	390	2	3	6	712	725
1	9	10	191	202	2	0	13	239	240	2	3	7	207	208
1	9	12	244	236	2	0	14	374	380	2	3	8	126	128
1	9	13	146	137	2	0	15	238	228	2	3	9	167	174
1	9	16	148	65	2	0	17	149	138	2	3	10	197	208
1	9	17	127	113	2	0	18	155	132	2	3	12	226	234
1	10	0	367	378	2	0	19	135	154	2	3	13	182	183
1	10	1	169	191	2	1	0	1065	1078	2	3	16	246	255
1	10	2	160	169	2	1	1	441	434	2	3	17	222	220
1	10	3	155	149	2	1	2	472	477	2	3	18	135	113
1	10	4	120	122	2	1	3	124	123	2	4	0	155	156
1	10	5	221	216	2	1	4	250	255	2	4	1	513	515
1	10	7	131	142	2	1	5	155	155	2	4	2	616	619
1	10	8	198	204	2	1	6	380	392	2	4	3	323	328
1	10	9	156	161	2	1	8	302	304	2	4	4	126	140
1	10	12	146	151	2	1	9	327	337	2	4	5	254	248
1	11	0	253	249	2	1	10	212	208	2	4	6	173	178
1	11	1	110	106	2	1	11	144	112	2	4	8	261	264
1	11	2	111	106	2	1	12	402	412	2	4	9	180	190
1	11	3	138	142	2	1	13	252	259	2	4	10	295	304
1	11	9	144	113	2	1	14	287	282	2	4	11	395	399
1	11	10	156	141	2	1	15	132	130	2	4	12	285	288
1	11	12	169	168	2	1	16	116	114	2	4	14	206	197
1	12	0	169	178	2	1	17	131	143	2	4	15	220	129
1	12	1	118	119	2	2	0	817	821	2	4	18	120	106
1	12	3	161	164	2	2	1	517	511	2	5	0	91	92
1	12	5	146	146	2	2	2	582	572	2	5	1	268	275
1	12	10	161	150	2	2	3	198	199	2	5	2	342	333
1	12	11	118	86	2	2	4	511	513	2	5	3	519	515
1	12	14	141	129	2	2	5	174	174	2	5	4	306	304
1	13	1	123	128	2	2	6	384	384	2	5	5	423	419
1	13	3	100	92	2	2	7	496	502	2	5	6	161	152
1	13	4	119	118	2	2	8	277	279	2	5	7	317	313
1	13	6	121	139	2	2	9	215	214	2	5	8	300	301
1	13	7	110	101	2	2	10	92	64	2	5	9	197	187
1	13	8	161	166	2	2	11	207	197	2	5	10	246	253
1	14	3	116	122	2	2	12	307	302	2	5	13	217	215
2	0	0	658	661	2	2	14	134	146	2	6	1	416	407
2	0	1	646	671	2	2	15	177	137	2	6	2	119	117
2	0	2	197	203	2	2	16	96	90	2	6	3	252	247
2	0	3	97	104	2	2	20	99	66	2	6	4	210	214
2	0	4	703	703	2	3	0	192	193	2	6	5	184	187
2	0	5	605	617	2	3	1	638	632	2	6	6	148	155

Table III (continued)

VALUES OF 10*FOBS & 10*FCALC FOR N3P3(H)CL4(ISO-C3H7)

H	K	L	FOBS	FCALC	H	K	L	FOBS	FCALC	H	K	L	FOBS	FCALC
2	6	7	298	300	2	10	11	181	174	3	2	7	305	312
2	6	8	411	421	2	11	1	121	123	3	2	8	137	144
2	6	9	242	250	2	11	2	140	148	3	2	9	267	270
2	6	10	223	224	2	11	4	186	194	3	2	10	308	302
2	6	11	242	252	2	11	5	153	154	3	2	11	113	146
2	6	13	118	137	2	11	8	180	196	3	2	14	115	97
2	6	14	113	120	2	11	9	131	133	3	2	16	188	183
2	6	19	109	122	2	11	10	139	142	3	2	17	130	119
2	7	0	116	110	2	12	0	112	134	3	2	18	123	130
2	7	2	148	145	2	12	1	116	104	3	3	0	352	358
2	7	3	465	468	2	12	3	216	223	3	3	1	288	282
2	7	4	336	322	2	12	8	110	140	3	3	2	792	802
2	7	5	266	283	2	12	10	109	83	3	3	3	98	113
2	7	6	324	335	2	15	4	125	102	3	3	5	79	71
2	7	7	332	331	3	0	1	389	383	3	3	8	197	197
2	7	11	198	198	3	0	2	181	179	3	3	9	209	252
2	7	16	135	126	3	0	3	184	188	3	3	10	159	149
2	7	17	171	171	3	0	4	390	397	3	3	14	191	194
2	8	1	202	200	3	0	5	359	361	3	3	15	102	94
2	8	2	192	214	3	0	6	91	82	3	3	16	105	104
2	8	3	290	302	3	0	7	484	505	3	3	18	100	70
2	8	4	210	218	3	0	9	179	173	3	4	0	202	204
2	8	5	384	394	3	0	10	105	130	3	4	1	175	177
2	8	6	195	194	3	0	11	488	478	3	4	2	199	197
2	8	7	156	174	3	0	12	163	167	3	4	3	400	407
2	8	9	226	240	3	0	13	187	179	3	4	4	363	358
2	8	14	117	107	3	1	0	270	269	3	4	5	395	402
2	8	17	138	118	3	1	1	155	162	3	4	6	270	262
2	9	0	190	182	3	1	2	284	285	3	4	7	444	453
2	9	1	293	298	3	1	3	571	570	3	4	9	335	335
2	9	3	199	213	3	1	4	275	269	3	4	10	127	116
2	9	4	240	255	3	1	5	526	527	3	4	13	121	138
2	9	5	235	237	3	1	6	296	307	3	4	16	183	178
2	9	6	176	180	3	1	7	82	91	3	5	0	377	384
2	9	7	166	160	3	1	8	236	241	3	5	1	174	177
2	9	8	200	218	3	1	9	522	545	3	5	2	463	471
2	9	9	156	127	3	1	11	275	282	3	5	3	526	536
2	9	12	97	97	3	1	12	103	120	3	5	4	204	201
2	9	13	113	111	3	1	17	99	69	3	5	5	93	97
2	9	14	101	107	3	1	19	112	115	3	5	7	102	110
2	9	16	114	100	3	2	0	644	660	3	5	8	139	127
2	10	1	199	209	3	2	1	91	67	3	5	9	292	290
2	10	2	144	142	3	2	2	285	287	3	5	10	273	276
2	10	6	209	211	3	2	3	475	487	3	5	11	358	360
2	10	8	122	145	3	2	4	758	778	3	5	12	129	135
2	10	9	227	213	3	2	5	354	349	3	5	15	95	65
2	10	10	131	127	3	2	6	98	97	3	6	0	610	617

Table III (continued)

VALUES OF 10*FOBS & 10*FCALC FOR N3P3(H)CL4(ISO-C3H7)

H	K	L	FOBS	FCALC	H	K	L	FOBS	FCALC	H	K	L	FOBS	FCALC
-	-	-	----	-----	-	-	-	----	-----	-	-	-	----	-----
3	6	1	124	138	3	14	4	118	101	4	4	4	157	156
3	6	2	363	358	3	14	11	218	67	4	4	6	156	116
3	6	4	340	356	4	0	1	564	586	4	4	7	213	206
3	6	5	137	143	4	0	2	192	189	4	4	8	157	161
3	6	7	232	225	4	0	3	201	200	4	4	10	155	161
3	6	8	162	156	4	0	4	194	183	4	4	12	258	271
3	6	9	196	216	4	0	6	173	174	4	5	1	290	284
3	6	10	97	99	4	0	7	273	276	4	5	4	213	203
3	6	11	118	151	4	0	8	185	207	4	5	8	192	197
3	6	12	172	186	4	0	10	189	192	4	5	10	175	176
3	6	13	168	160	4	0	13	121	116	4	5	12	193	191
3	6	16	150	141	4	0	18	109	123	4	5	13	145	151
3	6	17	113	96	4	1	0	238	238	4	6	1	299	296
3	7	1	146	151	4	1	1	268	271	4	6	2	142	143
3	7	2	288	298	4	1	3	245	266	4	6	3	138	129
3	7	4	288	297	4	1	4	173	178	4	6	4	200	198
3	7	5	270	272	4	1	5	235	231	4	6	8	120	129
3	7	6	203	204	4	1	6	312	311	4	6	9	95	72
3	7	8	135	133	4	1	7	197	182	4	6	10	261	192
3	7	9	137	140	4	1	8	217	210	4	6	11	187	183
3	7	11	97	120	4	1	9	115	81	4	6	12	100	70
3	8	1	198	195	4	1	10	128	117	4	6	13	112	109
3	8	2	222	233	4	1	16	116	92	4	7	0	153	152
3	8	4	407	406	4	1	17	129	105	4	7	3	161	165
3	8	7	114	85	4	2	2	246	240	4	7	5	90	91
3	8	11	113	113	4	2	3	226	222	4	7	6	260	276
3	8	12	143	144	4	2	4	266	268	4	7	8	140	154
3	8	14	199	115	4	2	5	185	202	4	7	9	97	76
3	8	16	188	181	4	2	8	241	229	4	7	10	161	132
3	9	3	229	234	4	2	9	190	175	4	8	0	121	135
3	9	4	186	193	4	2	10	100	111	4	8	3	98	89
3	9	7	174	174	4	2	12	102	102	4	8	5	112	108
3	9	9	203	185	4	3	0	318	316	4	8	7	155	140
3	10	1	119	101	4	3	1	263	272	4	8	8	179	168
3	10	3	167	176	4	3	2	99	75	4	9	2	145	136
3	10	5	231	242	4	3	3	215	233	4	9	3	175	167
3	10	6	216	201	4	3	5	434	457	4	9	5	131	151
3	10	7	131	142	4	3	6	282	283	4	9	9	152	159
3	10	9	223	233	4	3	7	337	340	4	10	0	210	211
3	11	1	187	179	4	3	10	140	149	4	10	1	101	123
3	11	2	204	202	4	3	11	95	91	4	10	2	127	137
3	11	3	166	154	4	3	14	211	194	4	10	4	100	108
3	12	0	194	185	4	3	16	115	105	4	11	1	123	113
3	12	4	186	196	4	4	0	184	189	4	11	5	126	145
3	12	7	196	46	4	4	1	241	242	4	12	3	115	88
3	13	5	126	136	4	4	2	327	324	5	0	3	284	287
3	13	7	143	162	4	4	3	244	247	5	0	4	209	200

Table III (continued)

VALUES OF 10*F0BS & 10*FCALC FOR N3P3(H)CL4(ISO-C3H7)

[illegible]

Table IV

Distances and Angles in 1,1-N₃P₃Cl₄(i-Pr)H, (VI).

<u>Distance</u> Å		<u>Angle</u> °	
P(1)-H(1)	1.263(7)	H(1)-P(1)-C(1)	108.87(2.58)
P(1)-C(1)	1.765(1)	P(1)-C(1)-C(2)	112.02(70)
P(1)-N(2)	1.614(6)	P(1)-C(1)-C(3)	109.77(79)
P(1)-N(6)	1.606(6)	C(2)-C(1)-C(3)	112.16(90)
		H(1)-P(1)-N(2)	101.25(2.87)
P(3)-N(1)	1.553(6)	H(1)-P(1)-N(6)	110.29(2.76)
P(3)-N(4)	1.575(6)	C(1)-P(1)-N(2)	110.43(37)
P(3)-Cl(1)	1.988(3)	C(1)-P(1)-N(6)	109.99(44)
P(3)-Cl(2)	1.992(3)	N(2)-P(1)-N(6)	115.57(30)
		P(1)-N(2)-P(3)	121.70(40)
P(5)-N(4)	1.581(5)	N(2)-P(3)-N(4)	120.08(30)
P(5)-N(6)	1.548(6)	P(3)-N(4)-P(5)	119.42(36)
P(5)-Cl(3)	1.997(4)	N(4)-P(5)-N(6)	120.00(35)
P(5)-Cl(4)	1.989(3)	P(5)-N(6)-P(1)	122.48(37)
		C1(1)-P(3)-Cl(2)	99.76(14)
C(1)-H(11)	0.870(76)	C1(3)-P(5)-Cl(4)	100.35(13)
C(1)-C(2)	1.540(15)	C1(1)-P(3)-N(2)	109.20(29)
C(1)-C(3)	1.537(15)	C1(2)-P(3)-N(2)	109.36(25)
		C1(1)-P(3)-N(4)	107.21(25)
C(2)-H(21)	0.998(78)	C1(2)-P(3)-N(4)	108.83(30)
C(2)-H(22)	1.044(69)	C1(3)-P(5)-N(4)	108.50(33)
C(2)-H(23)	1.105(87)	C1(4)-P(5)-N(4)	108.29(26)

Table IV (continued)

<u>Distance</u> \AA		<u>Angle</u> $^{\circ}$	
C(3)-H(31)	0.914(84)	Cl(3)-P(5)-N(6)	109.36(34)
C(3)-H(32)	0.896(73)	Cl(4)-P(5)-N(6)	109.03(27)
C(3)-H(33)	1.155(72)		

Table V

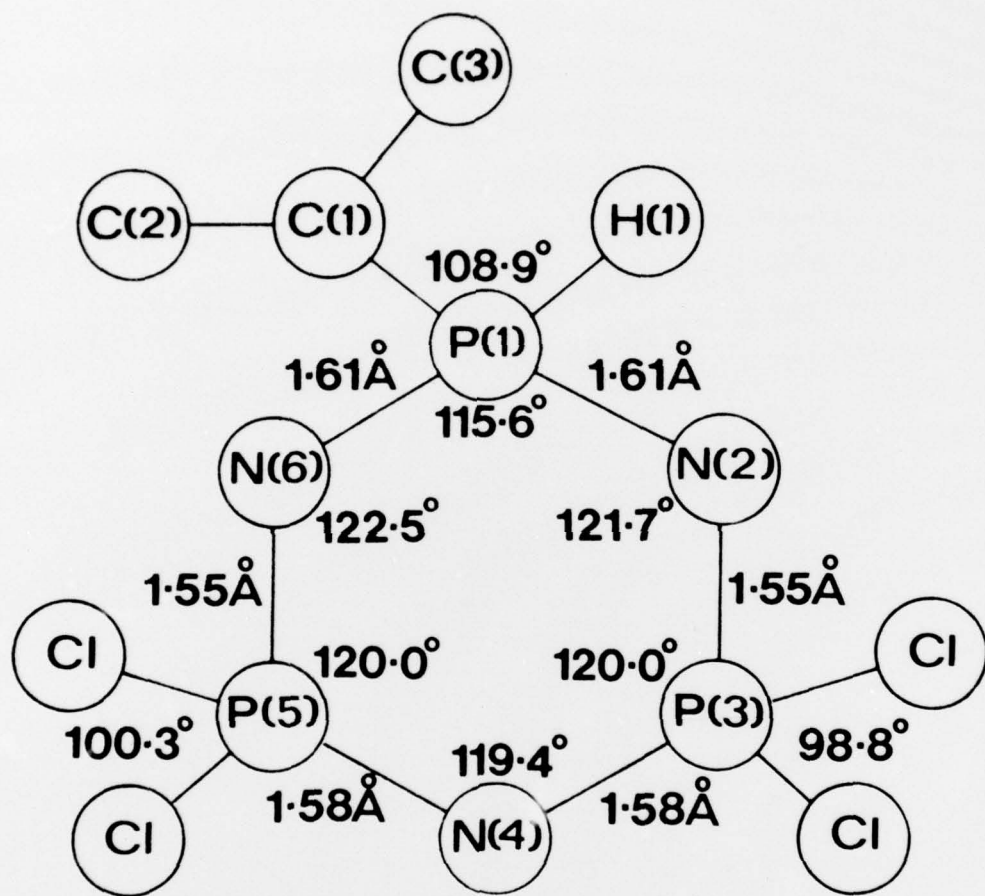
Compound	P-C bond, Å	C-P-C	X-P-X ^a	N-P-N ^b	Ref.
1,1-N ₃ P ₃ Cl ₄ (<u>1-Pr</u>)H	1.76	108.87°	100.05°	115.6°	this work
1,1-N ₃ P ₃ Cl ₄ (C ₆ H ₅) ₂	1.79	104.4°	100.30°	115.2°	17
1,1,3,3-N ₃ P ₃ Cl ₂ (C ₆ H ₅) ₄	1.79	104.4°	98.50°	115.0°, 116.0°	18
1,1-N ₄ P ₄ F ₆ (CH ₃) ₂	1.79	105.7°	94.10°	116.9°	19, 20
1,1,5,5-N ₄ P ₄ F ₄ (CH ₃) ₄	1.81	106.9°	98.50°	117.5°	19, 21

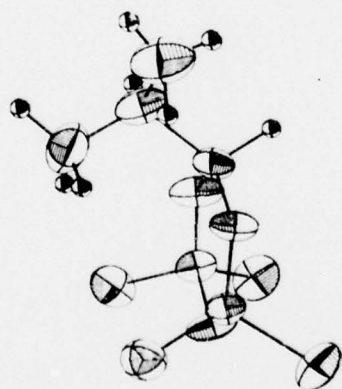
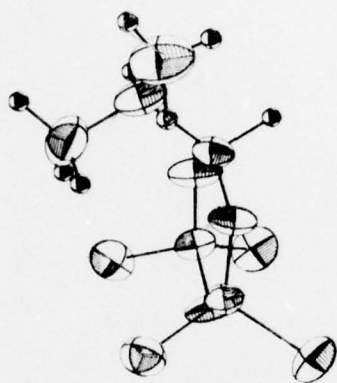
^a X = Chlorine or fluorine, exocyclic bond angle at halogenated phosphorus atoms.

^b endocyclic bond angle at the alkylated phosphorus atom.

Figure 1. Atom designations, selected bond angles and interatomic distances for $1,1\text{-N}_3\text{P}_3\text{Cl}_4(\underline{i}\text{-Pr})\text{H}$ (VI).

Figure 2. A stereoview of $1,1\text{-N}_3\text{P}_3\text{Cl}_4(\underline{i}\text{-Pr})\text{H}$, (VI).





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